**Compound 2.** A solution of 1 (1.00 g, 5.04 mmol) in dry THF (10 mL), pyridine (10 mL), and water (3 mL) was cooled to 0 °C, treated with osmium tetroxide (0.13 g, 0.50 mmol) and NMO (1.48 g, 12.6 mmol), and stirred overnight with slow warming to room temperature. At this point, sodium sulfite (2 g) was added, stirring was continued for 1 h, and dilution was made with CH₂Cl₂ (50 mL). The aqueous phase was evaporated to dryness and triturated with CH₂Cl₂. The combined organic layers were dried and evaporated. Chromatography of the residue on silica gel gave 2 (1.08 g, 93%) as a colorless oil; ¹H NMR (300 MHz, CDCl₃) δ 4.79 (br s, 1 H), 4.67 (d, J = 6.7 Hz, 1 H), 4.56 (d, J = 6.7 Hz, 1 H), 4.50 (d, J = 5.1 Hz, 1 H), 4.17 (br s, 1 H), 4.01 (t, J = 5.4 Hz, 1 H), 3.48 (br s, 1 H), 3.33 (s, 3 H), 2.39-2.27 (m, 1 H), 2.17-2.03 (m, 1 H), 1.95-1.84 (m, 1 H), 1.80-1.59 (series of m, 3 H); ¹³C NMR (75 MHz, CDCl₃) δ 176.5, 95.7, 95.5, 82.2, 70.4, 69.6, 55.7, 29.3, 29.1, 18.7; EI MS m/z (M⁺) calcd 232.0947, obsd 232.0925.

**Compounds 5 and 6.** A solution of 2 (0.26 g, 1.11 mmol) in dry CH₂Cl₂ (50 mL) was cooled to -78 °C under N₂, treated dropwise via syringe with a solution of Dibal-H in hexanes (8.0 mL of 1 M, 8.0 mmol), and stirred at this temperature for 1.5 h prior to the addition of acetic anhydride (10 mL). After 2 h, pyridine (12.1 mL) was introduced and the mixture was stirred overnight with slow warming to room temperature. A saturated solution of Rochelle's salt (20 mL) was added and one hour later the product was extracted in CH₂Cl₂, dried, and concentrated to furnish 3 as a clear oil (0.40 g, 61%) that was used directly.

Triacetate 3 (0.10 g, 0.27 mmol) and persilylated thymine (0.14 g, 0.50 mmol) were dissolved in THF (25 mL) at 0 °C, trimethylsilyl triflate (110 mg, 0.50 mmol) was added, and the reaction mixture was stirred at room temperature overnight prior to quenching with 1 M NaHCO₃ and extraction with ethyl acetate. The organic phase was washed with brine, dried and evaporated to give 28% of 4, 15% of 5 and 34% of 6 after chromatographic purification. These yields varied as a function of reaction conditions.
and particularly the purity of the trimethylsilyl triflate. Compound 4 proved to be labile to chromatography and was not fully characterized.

For 5: white solid, mp 195-199 °C; 1H NMR (300 MHz, CDCl₃) δ 8.70 (s, 1 H), 7.30 (d, J = 1.1 Hz, 1 H), 5.95 (d, J = 6.6 Hz, 1 H), 5.75-5.70 (m, 2 H), 4.21 (t, J = 7.5 Hz, 1 H), 2.17 (s, 3 H), 2.16-2.00 (m, 4 H), 1.93 (s, 3 H), 1.83-1.57 (m, 5 H), (OH proton missing); 13C NMR (75 MHz, CDCl₃) δ 169.9, 169.7, 163.3, 150.6, 136.4, 111.8, 94.0, 87.2, 78.3, 72.9, 71.0, 31.4, 30.2, 20.6, 20.5, 18.2, 12.6; ES MS m/z (M + Na)⁺ calcd 405.1274, obsd 405.1271.

For 6: white solid, mp 112-117 °C; IR (CHCl₃, cm⁻¹) 3390, 1751, 1695; 1H NMR (300 MHz, C₆D₆) δ 10.70 (s, 1 H), 10.37 (s, 1 H), 6.60 (s, 1 H), 6.57 (s, 1 H), 6.19 (d, J = 5.2 Hz, 1 H), 6.14-6.10 (m, 1 H), 5.76 (d, J = 6.4 Hz, 1 H), 5.17 (d, J = 10.6 Hz, 1 H), 4.56-4.53 (m, 1 H), 4.48 (d, J = 10.6 Hz, 1 H), 1.97 (s, 3 H), 1.80-1.60 (m, 15 H); 13C NMR (75 MHz, C₆D₆) δ 169.4, 169.1, 165.0, 164.3, 152.4, 150.8, 139.0, 137.8, 112.2, 111.4, 94.4, 90.2, 83.6, 74.7, 73.0, 72.0, 32.4, 31.9, 20.8, 20.1, 20.0, 12.4 (2C); ES MS m/z (M + Na)⁺ calcd 543.1705, obsd 543.1735.

**Compound 7.** A solution of 2 (0.16 g, 0.69 mmol) in CH₂Cl₂ (5.0 mL) containing DMAP (10 mg) was cooled to -40 °C and treated sequentially with acetic anhydride (3.0 mL, 32 mmol) and pyridine (4.0 mL, 49 mmol). The reaction mixture was allowed to warm to room temperature after 3 h, quenched with saturated NaHCO₃ solution (30 mL), and extracted with in CH₂Cl₂ (2 x 20 mL). The combined organic phases were washed with 1M HCl (20 mL) and brine (20 mL), then dried and evaporated. Chromatography of the residue on silica gel (elution with 3:2 petroleum ether-ether) gave 100 mg (48%) of 7 as a colorless oil; 1H NMR (300 MHz, CDCl₃) δ 5.92 (d, J = 5.3 Hz, 1 H), 5.77 (d, J = 5.3 Hz, 1 H), 4.68 (d, J = 6.8 Hz, 1 H), 4.60 (d, J = 6.8 Hz, 1 H), 4.05 (t, J = 5.8 Hz, 1 H), 3.38 (s, 3 H), 2.20-2.08 (m, 7 H), 2.06-1.98 (m, 2 H), 1.80-1.70 (m, 3 H); 13C NMR (75 MHz, CDCl₃) δ 170.2, 169.5, 168.7, 95.1, 93.6, 81.6, 70.4, 67.7, 55.8, 29.8, 29.0, 20.2, 19.9, 18.6; ES MS m/z (M + Na)⁺ calcd 339.1056, obsd 339.1048.
**Compound 9.** A solution of 7 (100 mg, 0.32 mmol) in CH₂Cl₂ (5.0 mL) containing 8 (160 mg, 0.57 mmol) was treated with trimethylsilyl triflate (0.16 mL, 0.83 mmol). After 5 min, triflic acid (0.02 mL, 0.23 mmol) was introduced. Five minutes later a white precipitate formed and the reaction mixture was quenched by the addition of saturated NaHCO₃ solution (15 mL). The separated aqueous layer was extracted with CH₂Cl₂ (3 x 10 mL), and the combined organic phases were washed with brine (10 mL), dried, and evaporated. Column chromatography of the residue on silica gel (elution with 100% ethyl acetate) returned 40 mg of unreacted 7 and furnished 20 mg (25%) of 9 as a white solid; ¹H NMR (300 MHz, CDCl₃) δ 8.38 (s, 1 H), 8.13 (s, 1 H), 5.72-5.64 (m, 4 H), 4.10-4.06 (m, 1 H), 2.12 (s, 3 H), 2.11 (s, 3 H), 2.09-1.98 (m, 3 H), 1.80-1.68 (m, 3 H) (NH₂ protons not observed); ¹³C NMR (125 MHz, CDCl₃) δ 169.7, 169.6, 169.0, 152.5, 149.4, 146.8, 142.3, 119.1, 93.3, 84.4, 71.9, 70.0, 67.6, 30.3, 29.2, 20.4, 20.1, 19.1; ES MS m/z (M + H)+ calcd 420.1519, obsd 420.1502.

**Compound 10.** To a solution of the butenolide (118 mg, 0.43 mmol) in CH₂Cl₂ (3 mL) at -10 °C was added potassium permanganate (99 mg, 0.65 mmol) and dicyclohexyl-18-crown-6 (11 mg, 0.03 mmol). After 4 h of stirring, sodium bisulfite (0.5 g) was introduced along with water (3 mL). Following extraction with CH₂Cl₂ (5x), the combined organic phases were dried and concentrated. The residue was chromatographed on silica gel (elution with 3:2 ether/hexanes) to give the lactol as an amorphous white solid (56 mg, 42%); ¹H NMR (300 MHz, CDCl₃) δ 7.17 (d, J = 8.5 Hz, 2 H), 6.87 (d, J = 8.5 Hz, 2 H), 4.60 (d, J = 11.8 Hz, 1 H), 4.53-4.49 (m, 2 H), 4.29 (d, J = 11.4 Hz, 1 H), 3.81-3.80 (m, 4 H), 3.20-2.75 (br s, 2 H), 2.10-2.02 (m, 1 H), 1.95-1.78 (m, 1 H), 1.76-1.65 (m, 4 H); ¹³C NMR (75 MHz, CDCl₃) δ 176.5, 164.1, 159.4, 129.3 (2C), 113.9 (2C), 95.9, 83.6, 71.2, 70.3, 69.7, 55.3, 29.4, 28.8, 18.9; EI MS m/z (M⁺) calcd 308.1260, obsd 308.1232; [α]D²² - 64.3 (c 1.0, CHCl₃).

The above lactone (170 mg, 0.56 mmol) was dissolved in CH$_2$Cl$_2$ (10 mL), cooled to -78 °C, treated dropwise during 10 min with Dibal-H (3.3 mL of 1 M in hexanes, 3.3 mmol), and left to stir for 3 h in the cold. Acetic anhydride (1 mL) was introduced, and the reaction mixture was maintained at -78 °C for an additional 2 h before being allowed to warm to room temperature and freed of CH$_2$Cl$_2$ under reduced pressure. More acetic anhydride (1 mL) was added followed by pyridine (2 mL). After 10 h of agitation, the reaction mixture was diluted with CH$_2$Cl$_2$ and poured carefully into 1 M NaHCO$_3$ solution. The separated organic phase was washed with water, 10% HCl, water, saturated CuSO$_4$ solution, and brine prior to drying and solvent evaporation.

Chromatography of the residue on silica gel provided 10 as a colorless oil (147 mg, 60% of a 4:1 mixture of anomers); $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.25-7.22 (m, 2 H), 6.86-6.81 (m, 2 H), 6.24 (d, $J = 4.8$ Hz, 0.2 H), 6.07 (d, $J = 2.3$ Hz, 0.8 H), 5.71 (d, $J = 4.9$ Hz, 0.8 H), 5.67 (d, $J = 5.9$ Hz, 0.2 H), 5.39 (dd, $J = 2.3$, 4.8 Hz, 0.8 H), 5.30 (t, $J = 5.1$ Hz, 0.2 H), 4.52-4.49 (m, 1 H), 4.36-4.30 (m, 1 H), 3.76 (s, 2.4 H), 3.75 (s, 0.6 H), 3.73-3.66 (m, 1 H), 2.25-1.60 (series of m, 15 H); $^{13}$C NMR (75 MHz, CDCl$_3$) (major isomer) $\delta$ 169.5, 169.4, 169.2, 159.1, 130.0, 129.2 (2C), 113.7 (2C), 97.9, 94.8, 84.1, 75.6, 71.5, 70.5, 55.1, 31.4, 28.8, 20.8, 20.4, 20.3, 20.0; (minor isomer) $\delta$ 169.6, 169.2, 169.1, 159.0, 129.9, 129.0 (2C), 113.7 (2C), 94.6, 93.2, 83.9, 70.8, 70.7, 69.5, 55.1, 30.7, 28.0, 21.0, 20.4, 20.1, 18.6; El MS m/z (M$^+$) calcd 436.1733, obsd 436.1745.

**Compound 11.** To a flask containing persilylated cytosine (237 mg, 1.19 mmol) was added a solution of 10 (103 mg, 0.2 mmol) in 1,2-dichloroethane (2 mL). Trimethylsilyl triflate (0.2 mL, 1.2 mmol) was next introduced, and the suspension became a clear, yellowish solution. The reaction mixture was stirred overnight, quenched with saturated NaHCO$_3$ solution, and extracted with ether. The combined organic phases were dried and freed of solvent to leave a residue, chromatography of which on silica gel (elution with 7:1 chloroform/methanol) gave 50 mg (60%) of 11 as a colorless oil. The anomeric ratio was 9:1 $\beta/\alpha$. For the major anomer: $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 7.37-
7.40 (m, 4 H), 6.89 (d, J = 6.0 Hz, 2 H), 6.24 (d, J = 5.8 Hz, 1 H), 5.77-5.67 (m, 2 H),
5.50 (t, J = 9.0 Hz, 1 H), 4.63 (d, J = 15.0 Hz, 1 H), 4.35 (d, J = 15.0 Hz, 1 H), 3.93-3.85
(m, 1 H), 3.78 (s, 3 H), 2.15-1.95 (m, 7 H), 1.90-1.55 (m, 5 H) (2 NH protons not
observed).

**Compound 12.** To a freshly prepared sample of persilylated uracil (344 mg, 1.34
mmol) was added a solution of 7 (103 mg, 0.2 mmol) in 1,2-dichloroethane (3 mL)
followed by trimethylsilyl triflate (0.21 mL, 1.34 mmol). The reaction mixture was
stirred overnight, poured into saturated NaHCO₃ solution, and extracted with CH₂Cl₂.
The combined organic phases were dried and concentrated. Chromatography of the
residue on silica gel (elution with 3:1 ether/hexanes) afforded 12 (68 mg, 54%) as a white
solid, mp 75-77 °C; ¹H NMR (300 MHz, CDCl₃) δ 9.03 (s, 1 H), 7.27 (m, 3 H), 6.91 (dd,
J = 2.0, 6.8 Hz, 2 H), 6.19 (d, J = 7.7 Hz, 1 H), 5.68 (d, J = 5.0 Hz, 1 H), 5.51 (d, J = 5.1
Hz, 1 H), 5.49 (d, J = 7.5 Hz, 1 H), 4.69 (d, J = 11.4 Hz, 1 H), 4.29 (d, J = 11.4 Hz, 1 H),
3.91 (t, J = 7.7 Hz, 1 H), 3.81 (s, 3 H), 2.17 (s, 3 H), 2.02 (s, 3 H), 1.78-1.59 (series of m,
4 H), 1.27-1.20 (m, 2 H); ¹³C NMR (75 MHz, CDCl₃) δ 169.7, 169.5, 162.7, 159.6,
150.7, 139.3, 129.6 (2C), 129.1, 114.1 (2C), 103.3, 93.4, 84.6, 83.9, 73.1, 71.4, 55.3,
30.0, 27.5, 20.6, 20.4, 17.9; El MS m/z (M⁺) calcd 488.1794, obsd 488.1814; [α]D²¹
-25.5 (c 1.10, CHCl₃).

**Compound 13.** A solution of 12 (70 mg, 0.14 mmol) in CH₂Cl₂ (1.2 mL) was
treated with DDQ (39 mg, 0.17 mmol) and 10 drops of water. After being stirred for 8 h,
the reaction mixture was diluted with CH₂Cl₂ (5 mL) and washed with 1 M NaHCO₃
solution. The aqueous phase was twice extracted with CH₂Cl₂, and the combined
organic layers were washed with brine and dried. Following concentration, the residue
was purified by chromatography on silica gel. Elution with ether gave the cyclopentanol
(47 mg, 91%) as a white solid, mp 129-131 °C; ¹H NMR (300 MHz, CDCl₃) δ 9.51 (s, 1
H), 7.58 (d, J = 8.2 Hz, 1 H), 6.08 (t, J = 3.2 Hz, 1 H), 5.79 (dd, J = 1.3, 8.1 Hz, 1 H),
5.68 (d, \( J = 3.1 \text{ Hz, 2 H} \)), 4.19 (m, 1 H), 3.42 (d, \( J = 3.8 \text{ Hz, 1 H} \)), 2.16 (s, 3 H), 2.12-1.98 (m, 2 H), 2.03 (s, 3 H), 1.82-1.53 (series of m, 4 H); \(^{13}\text{C NMR (75 MHz, CDCl}_3\)) \( \delta \)
170.0, 169.8, 163.3, 150.8, 140.8, 103.3, 94.3, 86.9, 78.1, 73.3, 71.2, 31.4, 30.2, 20.6, 20.4, 18.3; El MS \( m/z \) (M\(^+\)) calcd 368.1219, obsd 368.1208; \([\alpha]_{D}^{22}\) -14.8 (c 1.0 CHCl\(_3\)).

The above material (47 mg, 0.13 mmol) was dissolved in methanol (2 mL), treated with 5 mg of potassium carbonate, and stirred overnight. The solvent was evaporated and the residue was chromatographically purified (silica gel, elution with 5:1 chloroform/methanol). There was isolated 36 mg (99%) of 13, a white solid of mp 209 \(^\circ\text{C}; \(^1\text{H NMR (300 MHz, DMSO-d}_6\)) \( \delta \)
11.27 (br s, 1 H), 7.73 (d, \( J = 8.1 \text{ Hz, 1 H} \)), 5.81 (d, \( J = 7.8 \text{ Hz, 1 H} \)), 5.66 (d, \( J = 8.1 \text{ Hz, 1 H} \)), 5.30-5.23 (m, 1 H), 5.12 (d, \( J = 4.0 \text{ Hz, 1 H} \)), 5.09-5.05 (m, 1 H), 4.22-4.20 (m, 1 H), 4.02 (br s, 1 H), 3.87 (d, \( J = 3.8 \text{ Hz, 1 H} \)), 2.17-2.08 (m, 1 H), 1.89-1.82 (m, 1 H), 1.57-1.37 (m, 4 H); \(^{13}\text{C NMR (75 MHz, DMSO-d}_6\)) \( \delta \)
168.2, 156.3, 144.7, 106.2, 100.7, 90.0, 80.8, 77.6, 73.9, 35.4, 33.8, 22.0; El MS \( m/z \) (M\(^+\)- H\(_2\)O) calcd 266.0903, obsd 266.0907.